

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Application No. : 10/076,971

Confirmation No. 1426

Applicants

: Hamid Hojaji et al.

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DECLARATION UNDER 37 C.F.R. § 1.132

Mail Stop AMENDMENT Commissioner for Patents P.O. Box 1450 Alexandria, VA 22313-1450

Sir:

PEDRO M. BUARQUE DE MACEDO declares that:

- 1. He is a co-inventor of and is familiar with the present U.S. patent application Serial No. 10/076,971 filed on February 15, 2002 in the name of Hamid Hojaji and Pedro M. Buarque de Macedo and entitled "LARGE HIGH DENSITY FOAM GLASS TILE." He is also familiar with the Office Action dated September 22, 2004 issued therein and with the prior art references cited in the Office Action, including U.S. Patent No. 5,069,960 to Fukumoto et al. ("the '960 Patent").
- 2. He received a Bachelor of Science degree in Physics from George Washington University in Washington D.C. in 1959, and received a Ph.D. in physics from The Catholic University in 1963. From 1963 to 1967, he was employed with the National Bureau of Standards, and afterwards he continues to be associated with the National Bureau of Standards as a consultant. In 1967, he joined the department of mechanics at

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The Catholic University of America as an associate professor. In 1970, he became a codirector of the Vitreous State Laboratory and also a professor of chemical engineering and material science at the same university. Currently, he continues to be a co-director of the Vitreous State Laboratory and is a professor of physics at the same university. His primary area of expertise is in glass science research. In particular, he has developed technologies and products in the areas of fiber optics, defense fuels, and radioactive waste glass formulation. He has received 40 patents in the United States and many more worldwide, and has been noted as "the area's leading individual inventor in number of patents granted" by the January/February 1990 issue of *Washington Business Journal Magazine*. For more details of his background and areas of expertise, please refer to his curriculum vita attached hereto as Exhibit 1.

3. Under his direction and supervision, sample foam glass tiles were made in accordance with the teachings of the '960 Patent at the Vitreous State Laboratory of The Catholic University of America. Experiments were conducted to reproduce the three specific examples, Examples I, II, and III, described in the '960 Patent (See the '960 Patent, Col. 7, lines 1-68). As described in the '960 Patent, the same kind of vitrifiable mineral particles and the same foaming agent recipe were used in all of the three Examples, and the Examples differ from each other based on the type of coating used for the tile skin surface. All of the steps and recipes for producing foam glass tiles as prescribed by the '960 Patent were faithfully followed to every extent possible. The prescribed steps and recipes of the '960 Patent followed in the experiments as well as the diagrams of the equipments used therein were recorded in the Declarant's laboratory notebook, a copy of the relevant portion of which is attached hereto as Exhibit 2. To compact a mixture of the vitrifiable mineral particles and the effective amount of the foaming agent into tilelike blocks as specified by the '960 Patent (see Col. 4, lines 44-49), a compression device was constructed. (See Exhibit 2). The compacted vitrifiable mineral particles were coated (see Col. 4, line 62-Col. 5, line 3) and placed on a refractory bed, which allows air to flow between the tile and a firing stand. As the compacted vitrifiable mineral particles

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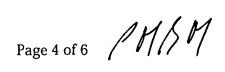
were subjected to the prescribed thermal treatment (*see* Col. 5, lines 21-48), they transformed into a thermally insulating foam glass tile and expanded linearly (by about a factor of 1.5) in size to a final size. This means that to produce a tile having a size of two feet by two feet, one needs to pressure-mold a 16 inches by 16 inches powdered material.

4. Initially, a small mold having a size of 2 inches by 2 inches was used to prepare an initial base material for reproducing thermally insulating foam glass tiles of the three Examples described in the '960 Patent. It was discovered that even the use of such a small mold required an enormous pressure of 2,500 to 3,000 pounds per square inches, or psi (equivalent to a compression force of 10,000 to 12,000 pounds) to achieve the initial density of 1.5 g/cm³ or 93 pcf before firing as disclosed in the '960 Patent (see Col. 6, line 46). Although the '960 Patent indicates that compression is needed to create molded tile-like block from a mixture of the vitrifiable mineral particles and the foaming agent (see Col. 4, lines 44-49), this surprisingly high level of pressure required to produce a foam glass tile in accordance with the teachings of the '960 Patent is not disclosed in the '960 Patent. The implication of this finding is that as the desired size of a foam glass tile increases, a compression force required to produce it may become enormous. For example, attempts were also made to produce foam glass tiles in accordance with the teachings of the '960 Patent from a 4.5 inches by 4.5 inches mold. If the same level of pressure used for the 2 inches by 2 inches mold were to be kept, a compression force of 50,625 to 60,750 pounds would have been required to produce foam glass tiles. However, the maximum compression force that the equipment used in the experiments was capable of applying reached only 24,000 pounds. Accordingly, the equipment used in the experiments could only apply a pressure of 1,200 psi to the 4.5 inches by 4.5 inches mold and could not apply the proper amount of pressure to produce the foam glass tiles in accordance with the teachings of the '960 Patent. According to the Declarant's calculations, production of a 2 feet by 2 feet tile having a weight greater than 30 pounds would require pressure-molding of a 16 inches by 16 inches powdered



materials in a mold under a compression force of 640,000 to 768,000 pounds (or 320 to 384 tons), if one were to follow the steps taught in the '960 Patent and apply the pressure of 2,500 to 3,000 psi. This amount of required compression force was clearly out of reach for the equipments used in the experiments. Declarant believes that such an enormous amount of required compression force would make the steps taught in the '960 Patent for producing large foam glass tiles meeting all the requirements of the present invention commercially impractical and prohibitively expensive.

5. Furthermore, in all of the reproductions of foam glass tiles produced in accordance with the Examples I, II, and III of the '960 Patent from the $2" \times 2"$ and the $4.5" \times 4.5"$ molds, large cracks and severe fractures were found near the lateral surfaces of the tiles. For each of the Examples, a number of re-runs of the experiments were attempted to eliminate those cracks and fractures, but such attempts were all unsuccessful. FIGS. 1-3 below show the cracks and fractures present in the reproduction of Examples I, II, and III of the '960 Patent, respectively. Declarant believes that those large cracks and fractures across the surfaces of the foam glass tiles produced in accordance with the teachings of the '960 Patent would make the tiles unsuitable for use as building surfaces and shock or impact absorber.





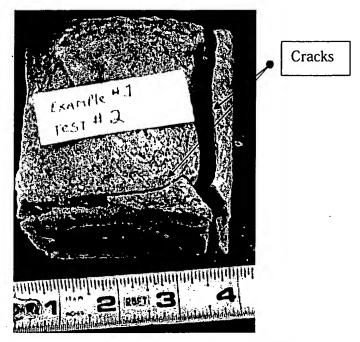


FIG. 1: Reproduction of Example I of the '960 Patent

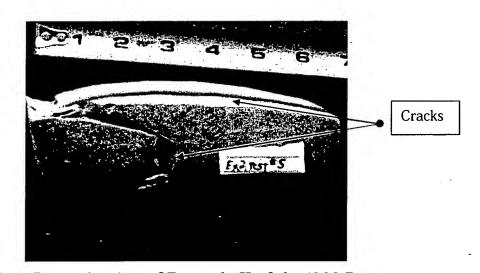


FIG. 2: Reproduction of Example II of the '960 Patent

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Appl. No. 10/076,971 Declaration dated Jan. 18, 2005 Reply to Office Action of Sept. 22, 2004



FIG. 3: Reproduction of Example III of the '960 Patent

- 6. In the Declarant's opinion, the results of the above-described experiments demonstrate that the '960 Patent does not teach or suggest as to how to produce foam glass tiles having a weight greater than 30 pounds that are suitable for building surfaces and shock absorption.
- 7. I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Date: January 18, 2005

By: <u>Feder M. Suagne de Macedo</u>, Ph.D.

Dr. Pedro B. Macedo

Vitreous State Laboratory, The Catholic University of America 405 Hannan Hall, 620 Michigan Ave, NE Washington, DC 20064 Tel: 202-319-5329, Fax: 202-319-4469, Email: macedo@cua.edu

Education

(1963) Ph.D. Physics, The Catholic University of America (1959) B.S. George Washington University

Publications

Over 100 referenced publications.

Over 40 issued patents in the U.S.A., and many more worldwide.

Employment

1963 – 1967 National Bureau of Standards—continued afterwards as a consultant.

1967 – Pres. Co-Director, Vitreous State Laboratory; Ordinary Professor (1970), Chemical Engineering and Material Sciences, currently Physics Department faculty, The Catholic University of America, Washington D.C. 20064

60's National Bureau of Standards recommended Dr. Macedo to an Academy of Science committee studying the technical problems with infrared transmitting glasses. He recognized that these problems were due to a lack in basic science. The proposal for the Themis Grant of DOD by The Catholic University of America was to form a center of excellence in Glass Science in order to address these problems. It was awarded and created the VSL in 1968. It resulted in Texas Instrument producing an infrared window within 18 months to look for the Vietcong from U2 spy planes.

70's VSL worked on fiber optics with the following results:

- a) U.S. Air Force Office of Scientific Research notified Congress that VSL's Fiber Optics research was their leading research effort (at which time Dr. Macedo's first patent application was submitted to the A.F.O.S.R.)
- b) The Fiber Optics patents were licensed to the following companies: Noranda, Pilkington, Sumitomo, American Cystoscope Makers, Biomedics International and Abbot.
- c) VSL worked for DARPA (DOD) on the development of the Cruise Missile. We significantly advanced the basic science of Lubrication leading to a new product by Monsanto, as well as very high energy fuels leading to a product produced by Shell.

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- 80's US GAO review found that VSL had the best record of technology transferred per R& D dollar invested, Some of VSL's best technologies for the decade are given below:
 - a) Patent on selective ion exchange which lead to the IPO of Duratek, Inc. (formerly GTS Duratek, Inc.).
 - b) Patent on Foam Glass which led to IPO of Energy Solutions.
 - c) Work on basic science of nuclear waste leaching resulted in Dr. Macedo being chairman of ASTM committee which provided the framework for permitting the fabrication of high level nuclear waste glass at Savannah River and West Valley.
 - d) Formulation of the high level nuclear waste glass permitted the cleanup of West Valley at a cost of \$1.5 Billion. This work included cooperation with the following companies: Westinghouse, DuPont, and Battelle.
 - e) Dr. P.B. Macedo is noted as "the area's leading individual inventor in number of patents granted in the last 10 years" (Washington Business Journal Magazine.
- 90's Dr Macedo's most successful patent, "The use of bubblers to stir and thereby increase throughput of Joule Heated Melters", resulted in:
 - a) The U.S. DOE notified Congress that the GTS Duratek/VSL team performed the most successful research that year.
 - b) GTS Duratek was awarded a privatization contract to vitrify $\sim 1,000$ tons of glass in M-Area of the DOE Savannah River Plant.
 - c) Permitted GTS Duratek to go public for a second time and obtain sufficient funds to increase annual sales from the mid \$20M to the mid \$200M.
 - d) GTS Duratek formed a partnership with BNFL that won the privatization contracts both part A and part B for the Hanford site tank cleanup project.
 - e) Additional companies which VSL cooperated in development and technology transfer during the 90's: Lockheed, Martin Marietta, Molten Metal, EG&G, B&W, Morrison &Knudsen, and Mason Hanger.
- 2000's The inventions of the 90's continue to work: Dr. Harry Boston, former manager of the DOE's Office of River Protection (which is the leading waste tank cleanup project at the Hanford site), stated "VSL/CUA's unique expertise will contribute to making the cleanup 20 years faster and up to \$20 billion cheaper."
 - a) Based on Duratek's design and VSL's research, Bechtel is building the vitrification plant for High Level Nuclear Waste for about \$5B at the Hanford site, called "...the most contaminated site in the western world".
 - b) Dr. Macedo has started a new line of research by writing three new patent applications in foam glass-Silica WebTM -"The More Responsible Alternative in Building Products" TM

Recent Patent Application Filings

Hojaji, Hamid et al., Large high density foam glass tile composite, U.S. Publ. No. 2003-0145534, published August 7, 2004 and U.S. Publ. No. 2004-0123535, published July 1, 2004.

Buarque de Macedo, Pedro M. et al., Chalcogenide ceramics for the disposal of radioactive and/or hazardous waste, U.S. Publ. No. 2004-0111003 A1, published June 10, 2004.

Hojaji, Hamid et al., Strong, High Density Foam Glass Tile, pending U.S. Patent Application filed 2003.

Buarque de Macedo, Pedro M., Prestressed Foam Glass Tiles, pending U.S. Patent Application, filed 2003.

Buarque de Macedo, Pedro M. et al., Method and Apparatus of Removing Antimony, pending U.S. Patent Application, filed 2004.

Copy of PATENT Request PAGE #7 Showing ExAMPLES

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EXAMPLE I

The natural vitrifiable mineral particles used were a mixture of 20 percent by weight of liparite of Japanese origin and 80 percent by weight of volcanic ash, the mixture having a chemical composition (in weight percent) of SiO2 - 75.4; Al2O3 - 12.2; Fe2O3 - 1.0; CaO - 0.8; K₂O - 4.2; Na₂O - 3.6; TiO₂ - 0.1; and trace amounts of MgO, with a loss on ignition of 2.9 weight percent. The vitrifying agent used was a borosilicate glass powder having a softening point of 585° C. and a chemical composition (in weight percent) of SiO2 - 65.9; Al2O3 - 4.1; B₂O₃ - 18.3; ZnÖ - 3.2; CaO - 1.5; MgO - 0.1; K₂O - 1.7; and Na2O - 5.2. Incidentally, it has been found that borosilicate glass is very useful because it substantially lowers the softening temperature of the vitrifiable mineral particles and the resultant mixture has substantially the same thermal expansion coefficient as the foam glass body formed from the mineral particles.

The mixture of the above-mentioned mineral particles was pulverized by crushing and milling into a powder having a mean particle size of about 15 microns, followed by addition thereto of 0.18 percent by weight of a silicon carbide foaming agent. The resulting mixture was compression molded into a 120×120×40 mm

thick tile-like block.

A virifying agent composition was prepared by milling together 100 parts by weight of the above-described borosilicate glass powder, 5 parts of quartz kaolin clay, and 70 parts of water for a period of 10 hours. The composition was applied by spray coating about 20 mg per square centimeter on a wet basis onto the top surface of the tile-like block. The coated block was fired in a tunnel kiln for a total of 24 hours, at a maximum temperature of 1180° C. for 1 hour, to produce a 180×180×60 mm thick foam glass tile 4 consisting of a foam glass body I and a surface skin 2, as shown in FIG. 1. The surface hardness and strength of the surface skin 2 were higher than those of the foam glass body 3. The 40 thermal shock resistance of the surface skin was substantially the same as that of the foam glass body.

EXAMPLE II

Example I was repeated, except that the vitrifying 45 agent composition was formulated from 18.9 parts of the borosilicate glass powder, 78.5 parts of the above-described vitrifiable mineral mixture containing no foaming agent, 1.3 parts of quartz kaolin clay, 1.4 parts of gypsum and 76.0 parts of water, all cm a weight basis. 50 The composition was applied by spray coating about 30 mg per square centimeter on a wet basis onto the top surface of the tile-like block. After furing as above, the strength and smoothness of the resulting surface skin 2 were better than those obtained in Example 1.

EXAMPLE III

Example I was again repeated, except that the vitrifying agent consisted of a water glass (aqueous sodium silicate No. 3 according to Japanese Industrial Stan-60 dards) having a chemical composition as follows (in weight percent): SiO₂ about 29; Na₂O about 10; Fe 0.02 or less; water insolubles 0.2 or less; and balance of water. The vitrifying agent was applied by spray coating about 37 mg per square centimeter on a wet basis onto 65 the top surface of the tile-like block. After firing as above, the resulting skin-surfaced foam glass tile was substantially the same as the tile obtained in Example I.

EXAMPLE IV

The materials used for the intermediate layer 12 of the foam glass tile 16, as illustrated in FIG. 2, were (A) the vitrifiable mineral particle mixture defined in Example I in combination with 0.01 weight percent of a SiC foaming agent; (B) the vitrifiable mineral particle mixture defined in Example I in combination with 0.03 weight percent of a SiC foaming agent; or (C) the vitrifiable mineral particle mixture as defined in Example I in combination with 0.06 weight percent of a SiC foaming agent.

The process of Example II was repeated, except that either intermediate layer material (A), (B) or (C) was spray-coated onto the tile-like block (120×120×40 mm) to form a coated layer approximately 2 mm thick. Thus, a skin-surfaced foam glass tile 16 was formed having dimensions of about 180×180×60 mm thick and consisting of a foam glass body 10, an intermediate layer

12 and a rigid surface skin layer 14.

The resulting skin-surfaced tiles (A), (B) and (C) exhibited no pinholes in the surface skin layers. The thermal shock test, evaluated by measuring the difference in temperature between the cooling water and the surface temperature of the foam glass tile when cracks occur, resulted in a temperature difference of 50° C. or higher in the case of tile (A), and a difference of 100° C. or more in the case of tiles (B) and (C). The tile obtained in Example II exhibited some pinholes in its surface skin, and the thermal shock test thereof resulted in a temperature difference of 100° C. or more.

What is claimed is:

- A thermally insulating foam glass tile comprising, a tile-like body consisting essentially of a first vitrifiable mineral and having a first volume of closed cells and
- a rigid reinforcing skin layer consisting essentially of a vitrified mixture of said first vitrifiable mineral and a vitrifying agent for said first vitrifiable mineral permeated into at least a top surface of said tile-like body.
- The foam glass tile as claimed in claim 1, wherein there is a gradual transition from said skin layer to said tile-like body.
- 3. The foam glass tile as claimed in claim 1 wherein said skin layer contains between about 3 mg/cm² and about 50 mg/cm² of said vitrifying agent on a dry basis.
- The foam glass tile as claimed in claim 1 wherein said skin layer has a thickness of between about 0.05 mm and about 1 mm.
- 5. The foam glass tile as claimed in claim 1, wherein said tile-like body has a predetermined coefficient of thermal expansion and said skin layer has a coefficient of thermal expansion substantially equal to said predetermined coefficient of thermal expansion.
- The foam glass tile as claimed in claim 1, wherein said vitrifying agent comprises a borosilicate glass powder.
- The foam glass tile as claimed in claim 1, wherein said tile-like body further includes a coloring pigment.

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8. The foam glass tile as claimed in claim 1 further comprising an intermediate layer integrally melt-bonded between said tile-like body and said skin layer, said intermediate layer consisting essentially of a second vitrifiable mineral and having a volume of closed cells less than said first volume.

Jonathan Stran 10-29-04

BOROSILICATE GLASS Rowder IN WEIGHT AR CENT

KZO 1.7 دم٥ 1.5 MgO 0.1

QUARTZ KAOLIN CLAY

H20

Chemical Composition IN WEIGHT PER CENT

> 5.0 70,0 Reviewed By:

MIX TOGETHER FOR TEN LOURS SPRAY DONTO.

THE FORM GIASS TITE body RECEIVES 2.88 grams of COATING (20 mg of 1 per sounde centimeter 1)

FIRING STAND: THE COATED FORM GLASS THE IS PLACED ON A FIRING STANDE. THE BASE of THE FIRING STAND IS INCOME! STEEL #601, YE" & 8" x8" southe FOUR INTER LOCKING SIDE PIECES, WITH MY INTERIOR SIZE OF 180x 180 x 60 MM TAIL ARE HELD TO THE BASE WITH Clips. REFRACTORY INORGANIC GRANVIES, HAVING A MEAN SIZE of \$ 16" OR 1.5 MM

THE Placed between THE FORM GLASS TILE AND FIRING STAND.

FIRING STAND WITH FRAM GIASS TILE, IS PIACED INTO A KILN FOR 24 LOURS AT A MAXIMUM TEMPERATURE of 1180's, FOR ONE HOUR TO PROduce A FILE 7"x7"x 23/8". DURATION of RAMP UP TEMPERATURE AND RAMP DOWN TEMPERATURES ARE PROGRAMMED INTO THE KILL CONTROller

forethan Stran 10-29-04

Jordan Stoon 10-29-04

EXAMPLE # 2

VSL-SWIOI p.76

(B) VITRIFIABLE MINERAL PARTICLES

30 % LIPARITE 80 % VOICANIC ASh

Chemical Composition in weight PER CENT

5:02	75.4°/0
A1203	iス.ス
KZO	٧. ٦
N ₁ QO	3.6
Fez03	1.0
CAO	0.8
TiOz	0.1
7190	TRACE

CRUSH and Mill THESE PARTICLES INTO A POWder which Passes MROUGH A 200 MESH Sieve

By weight 0.18% of A Silicon CARBIDE 0.18

MOLD, NOW CONSIDERED THE FORM GIASS TILE body

1 COATING

MAKE ANOTHER MIXTURE OF A
SET ASIDE 78.5 PARTS OF MIXTURE A) TO USE IN CONTING

MIX VITRIFYING AGENT BORDSILICATE GLASS

IN WRIGHT AR CENT

S.Oz 65.9%

Bzc3 18.3

MAZOD 5.2

AlzO3 4.1

ZNO 3.2

KzO 1.7

LaO 1.5

Myo 0.1

Reviewed By: $\frac{\mathcal{G}}{\mathcal{G}}$.

SET ASIDE 18.9 PARTS of BORDSILICATE GLASS MIXTURE, TO USE IN CONTING

MIXTURE (A) 78.5 PARTS
BORDSINICATE GLASS 18.9

OVANTZ KADIN CLAY 1.3

GYPSUM 1.4

H20 76.0

MIX COMPONENTS OF FOR 10 HOURS

SPRAY 4.32 GRAHS OF COATING (D) ONTO FORM GLASS TILE BODY (C)
PLACE COATED TILE ONTO PREPARED (AS IN EXAMPLE HI) FIRING STAND

6 PROGRAM RAMP UP TEMPERATURES AND DURATION INTO KIND CONTROller TO ATTAIN 11802 FOR 1 HOUR, AND RAMP DOWN TEMPERATURE, AND DURATION TO COMPLETE 24 HOUR CYCLE.

Example # 3

VSL-SWIDI P.77

@ V.TRIFIABLE MINICRAL PARTICLES:

20% LIPARITE 80% VOICANIC ASh

Chemical composition IN WEIGHT PER CENT

75.4 % 5:02 Alzos 12.2 **4.** 入 K20 NUZU 3.6 1.0 FezOs CuD 0.8 Tioz 01 TRACE Mgo

CRUSH AND Mill THESE PARTICLES INTO A POWER which Sieves below 200 MESH

B FOATTING AGENT By Weight, 0.18 % of A

Silicon CAMBIDE 0,18

MIX A+B. CUMPRESS INTO A 120 x 120 x 40 MM Mold

D COATING
WATERGIASS AS VITRIFYING AGENT
IN WEIGHT PER CENT

5:02 ± 29 % NG20 ± 10 Fe 0.02 H20 ± 60

Blend well

- E SPRAY (D ONTO (C)
 THE FOAM GLASS THE BODY RECEIVES 5.33 GRAMS OF COATING
 31 Mg AIR SQUARE CENTIMETER
- F Place control Tile onto PREPARED (AS IN EXAMPLE #1) FIRING STAND
- (FIRING STAND WITH FORM GIASS THE, IS PLACED INTO A KILN FOR 24 HOURS AT A MAXIMUM TEMPERATURE of 1180°C, FOR 1 hour to produce a tile 180 x 180 x 60 mm. Duration of RAMP up Temperature and RAMP Down Temperatures are programmed into the Kiln Controller.

Jonathan Stram 10-29-04

Reviewed By: 4.P. Dails: 12/14/0

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10-29-04 VSI-SW101 P. 79 Parce of Stor . THE SHIPS MAE CONTROL WITH A MOLE RETURNSE 50'07-013 (= g). PLECONIERS Receive D.E. To close GAF.

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